

PROCESS AND APPARATUS FOR PRODUCING METAL NANOPARTICLES

Field of the invention

5 The present invention relates to a novel process for producing metal nanoparticles by electro-explosion of wires. More particularly, the present invention provides a process for the production of nanoparticles comprising exploding wires into small fragments by application of suitable voltages, at low voltages in order to supply energy to the wires such that the nanoparticle size depends upon various controlling parameters such as applied voltages and current densities. The present invention
10 provides a simple, repeatable process to achieve large production volumes for nanoparticles.

Background of the invention

Nanoparticles have been a source of great interest due to their novel electrical, optical, physical, chemical, magnetic etc. properties. They have significant potential for
15 a wide range of applications including catalysis, magnetic recording media, optoelectronic materials, magnetic fluids, composite materials, fuel cells, pigments and sensors. Their uniqueness arises from their high ratio of surface area to volume (aspect ratio), as these materials have diameters of 100 nm or less.

There are several reports of physical/chemical processes for the production of
20 nanoparticles in technical literature. Nanoparticles prepared by physical methods such as vapour deposition and sputtering have high quality, i.e. clean surfaces and uniform particle size distribution. However, industrial applications for such particles are limited due to the low production rates and high cost. Alternative chemical production methods, such as thermal decomposition and precipitation are currently being studied
25 for the preparation of a wide range of nanoparticles. Chemical methods can provide large quantities of ceramic particles for industrial applications. However, except for precious metals, chemical methods are generally not applied to the production of metallic nanoparticles.

Reference is made to U.S. Patent 6,203,768 for "Process for the production of
30 ultrafine particles" by McCormick, Paul Gerard, Ding, Jun, Miao, Wie-Fang, Street, Robert; wherein a mixture of a metal compound and a suitable reagent is subjected to mechanical activation (using ball mills) to increase the reaction kinetics such that a chemical reaction can occur; which produces a solid nano phase substance. Here the particle size depends on ball size and milling time. The main drawback of this process

is that it is time consuming. In one example, for steel balls of size 4.8mm; 10nm size particles are produced by 8 hours milling.

Reference is made to U.S. Patent 5,772,754 "Ultra-fine particles and their production method thereof" by Tanaka, Shun-ichiro, Xu, Bing She; wherein electron beam is used to facilitate the reaction. By irradiating with an electron beam in vacuum of metastable particles of a metal oxide, dispersed on an amorphous carbon film, an ultrafine powder comprising ultrafine stable particles of metal oxide and nanoparticles of a metal are produced. The metal particles so produced is a component of the metal oxide. The main drawbacks of this process are that it has to be operated in a vacuum environment and the product is a mixture of the metal and its oxide.

Reference is made to U.S. Patent 4,503,013 "Ultra-high power exploding wire systems" by Lowther, Frank E., where explosion of wires is used to enhance chemical and physical reactions. The material to be exploded is mounted in the housing of a reactor and firing is provided in the housing which will explode the members when the element is moved into contact there with. Here firing is meant in the sense of providing the electrical power for exploding the material. Hot, fast metallic atoms from the exploding element activate given physical or chemical reactions. Here explosion is used to enhance the reaction rate and not to produce nanoparticles. The drawback of this process is that it is used to enhance reactivity and does not result in the formation of nanoparticles.

Reference is made here to US Patent Application 0020037320 of 2002, wherein it is claimed that nanomaterials are produced with the help of two electrodes by achieving sputtering from one of the electrodes (the target material) in the presence of a dense medium. The basic process, namely sputtering, is well-established in the literature. Use is made here of the process of sputtering in a dense medium to achieve large sputtering yields from a target material. The particles produced are claimed to be nanometer in size. The drawback of this process is that only an ionisable medium can be employed for this purpose together with the use of DC voltages high enough to achieve ionization of the medium. Further, only one of the electrodes is consumed in the process to produce the nanoparticles.

It is therefore important to devise a process for the production of nanoparticles which is simple to apply, results in large production volumes and is therefore industrially feasible, economical and energy efficient.

Objects of the invention

The main object of the present invention is to provide a novel process for producing metal nanoparticles by electro-explosion of wires, which obviates the above drawbacks.

Another object of the present invention is to produce nanoparticles from conducting parent materials shaped in the form of a wire / plate and are used as electrodes, whereby both electrodes produce the particles.

Yet another object of the present invention is to generate nanoparticles through the dominant mechanism of spark explosion, which is an adaptation of the phenomena called electro-explosion of wires.

Still another object of the present invention is to use low voltages between 12V–50V DC, while also having the possibility of employing AC voltages for the purpose, thereby reducing the energy costs involved in production of nanoparticles.

Still another object of the present invention is to provide for the use of various dense liquids as medium of explosion, irrespective of their dielectric property.

Still another object of the present invention is to provide a method by which capping is achieved of the nanomaterials with a suitable layer of an inert material produced around the nanomaterial due to a reactive step in the environment of the medium present.

Summary of the invention

The present invention provides a novel process for the production of metallic nanoparticles based on the electro-explosion of wires in a suitable medium. Particularly, the present invention relates to a novel process for the production of metallic nanoparticles by way of controlled explosion of the metal in a suitable medium applying voltages of about 12V and above, to two electrodes, one in the form of a wire and another in the form of a plate, so as to achieve a spark between the electrodes, thereby simulating a situation where the wire cross-section is pinched or reduced and whereby high current densities are achieved along the length of the wire, instantaneously exploding the electrodes by sending shock waves through the bulk of the material, melting them and dispersing them to form small fragments which are then collected by a suitable medium in which the process is initiated, not only for efficient recovery of the material but also to form a protective layer around the now highly reactive nanomaterial so to prevent the nanomaterials from coalescing into large particles by forming a cap, wherein the capping efficiency is determined by the combination of the metal and the medium in which the metal electrodes are exploded

and the particle size can be modified by way of altering parameters such as applied voltage, explosion current density and wire diameters.

Accordingly, the present invention provides a process for the production of metallic nanoparticles by the controlled electro-explosion of a metallic wire in a suitable medium comprising:

- i. applying a voltage of greater than 12V to a first electrode and a second electrode, both said first and said second electrodes being formed of the metal whose nanoparticles are desired, said first electrode being in the form of a plate, and said second electrode being in the form of a wire, so as to achieve a spark between the said first and second electrodes, thereby simulating a situation where the second electrode cross-section is pinched or reduced and whereby high current densities are achieved along the length of the second electrode,
- ii. instantaneously exploding both said first and second electrodes by sending shock waves through the bulk of the material, thereby melting the electrodes and dispersing them to form nanoparticles of the metal, collecting the fragments in a suitable medium such as herein described and recovering the nanoparticles therefrom.

The present also provides an apparatus for the production of metallic nanoparticles by the controlled electro-explosion of a metallic wire in a suitable medium which comprises a reaction vessel containing said medium, a first and second electrodes mounted inside said vessel, submerged in said medium, said first and second electrodes being formed of a metal whose nanoparticles are desired, said first electrode being in the form of a plate, and said second electrode being in the form of a wire, so as to achieve a spark between the said first and second electrodes, thereby simulating a situation where the second electrode cross-section is pinched or reduced and whereby high current densities are achieved along the length of the second electrode, said electrodes being connected to a power source so that current is passed through said electrodes, instantaneously exploding both said first and second electrodes by sending shock waves through the bulk of the material, thereby melting the electrodes and dispersing them to form said nanoparticles of said metal.

Preferably, said first electrode is mounted wherein said first electrode is mounted perpendicular to the base of said reactor.

In another preferred feature, said first electrode is mounted in said reaction vessel through stainless steel slides on an insulating block

In another preferred feature, said insulating block is a plastic block.

5 In yet another preferred feature, said second electrode is mounted in said reaction vessel through a guide. The guide is preferably, an "L" shaped glass tube, mounted through an insulating mounting means fixed in said reaction vessel. The "L" shaped glass tube is mounted on said insulating mounted means such that it collimates said second electrode, passing therethrough to strike said first electrode along its normal.

10 The nanomaterials that have been realized are independent particles, comparable in size to other nanomaterials reported in the prior art literature. The process is very energy intensive since only relatively low voltages are applied, and also results in high volumes of nanoparticles being produced since both electrodes are consumed.

15 Accordingly the present invention provides a novel process for producing metal nanoparticles by electro-explosion of wires which comprises placing a wire and a plate of the same material in a dense medium, preferably water, in such a manner so as to guide the wire in a straight line to make contact with the plate intermittently.

20 In an embodiment of the present invention a potential difference of 12V – 48V DC, preferably 36V DC, is applied between the plate and the wire so as to produce explosion when brought into contact with each other to achieve the desired nanoparticles.

25 In an embodiment of the present invention the cross-section of wires employed are in the range of $0.4411 \times 10^{-5} \text{ cm}^2$ - $1.7721 \times 10^{-5} \text{ cm}^2$ to carry current in the range of $0.96 \times 10^6 \text{ A/m}^2$ – $77.6 \times 10^6 \text{ A/m}^2$ to obtain the desired nanoparticles after explosion.

30 In yet another embodiment of the present invention all metals capable of maintaining a current density of 10^5 A/m^2 or higher is amenable to production of nanomaterials, whose size are in the range of a few nanometers, and whose examples are transition metals such as Fe and Cu, noble metals such as Ag and Group III metals such as Al.

Brief description of the accompanying drawings:

Figure 1 is a depiction of a Z-pinch obtained in a wire due to the self-inductive interactions between the circuit elements resulting in the development of an axial force. Figure 2 is a top view of the wire guide for carrying out the process of the invention.

Figure 3 is a front view of the wire guide for carrying out the process of the invention.

Figure 4 is a schematic view of the reaction vessel used for carrying out the process of the invention.

5 Figure 5 shows the AFM data collected for nano-crystalline copper particles allowing for the recording of particles in the range 27nm – 72nm.

Figure 6 shows the XRD data of bulk and nanoparticles of copper.

Figure 7 shows the AFM data collected for nano-crystalline silver particles allowing for the recording of particles in the range 50nm - 200nm.

Figure 8 shows the XRD data of bulk and nanoparticles of silver.

10 Figure 9 shows the AFM data collected for nano-crystalline iron particles allowing for the recording of particles in the range 10nm – 50nm.

Figure 10 shows the XRD data of bulk and nanoparticles of iron.

Figure 11 shows the AFM data collected for nano-crystalline aluminium particles allowing for the recording of particles in the range 40nm - 150nm.

15 Figure 12 shows the XRD data of bulk and nanoparticles of aluminium.

Detailed description of the invention

The present invention provides metal nanoparticles made by the novel process of the present invention as detailed below.

20 The exploding wire technique, EEW was basically the guiding principle behind the actual experimental arrangement to synthesise nanopowders of Cu, Fe, Al and Ag metals.

In this technique, very high currents are passed through thin metallic wires in very short time. Extraordinary temperatures (given by the kinetic energy of the electrons) are achieved as a result. The wire is converted into a plasma state. Super
25 cooled metal plasma expands with supersonic velocity, creating a shock wave in the ionised surrounding gas.

The key factors for the control of EEW:

- The current I should be very large, i.e. very high current density is required for the explosion process, which basically gives rise to some non-linearity in the
30 volt-ampere characteristics.
- The initial stage of EEW is a Z-pinch which essentially decreases the cross-sectional area of contact to 1/100th of the rest of the conductor, thereby tremendously increasing the current density which is required for explosion to take place, whenever contact was made and broken.

- The medium which is to be used for doing the explosion.

Due to self-inductive interactions between the circuit elements, an axial force develops which causes a Z-pinch in the wire (Figure 1). These compressive pinches apply radially inward pressure on the cylindrical wire. This forms a constriction in the wire. Due to high magnetic field energy density, the wire explodes in this region when high current is passed in a short period of time.

If two sharp edged wires are brought close to each other and a potential difference is applied between the two, then as soon as contact is made wire explosion takes place. Electrons, atoms and ions are scattered and photons are generated resulting in a plasma state. Photons are produced due to de-excitation and the explosion plumes of characteristic colour are observed.

Medium: In general an inert atmosphere is suitable as a medium, one which does not interact with the plasma formed during the explosion, e.g. butanol, heavy oils, etc. Oxygen or any other active medium is not suitable since with mobile carriers they form compounds.

Finally, wire explosion was done in water for various reasons:-

- a) Experiments already reported in the literature suggest that the initial stage of discharge is same in water and air.
- b) Water acts as a suitable medium for attenuating the speed of the metal ions during explosion, hence controls the entire procedure.
- c) Water is a good thermal conductor and therefore helps in temperature regulation of the process.
- d) It is easily accessible.
- e) After the collection of nanoparticles of the metal in water – some technique suitable for the medium used should be employed to separate them from it. This is easily possible by centrifuging the sample solution to collect the nanoparticles.
- f) Water also provides the necessary capping to prevent grain growth.

Use of wire and plate: In case of a Z-pinch two end-tapered metal rods were used for explosion. Same result can be achieved by a movable sharp-edged wire and a fixed plate as a target. This is in the form of the letter K and hence named K-pinch, was used to explode thin metal wires on a plate of the same metal such that the plate and the wire are attached to the opposite terminals of a battery in the circuit. A potential difference is maintained by the batteries between them while contact was made and

broken between the plate and wire. This resulted into an explosion. This has the following advantages:

(i) The tapered ends would have got blunt after a few explosions as the material gets exhausted. The cross-sectional area at the Z-pinch would change consequently and it would be necessary to taper them again. Instead, a wire of uniform cross-sectional area could be used by pushing in more and more each time the explosion consumed it.

(ii) The wire goes and touches the copper plate at a single point so indeed the requirement of confining the point of contact (the Z-pinch being modified to a K-pinch), hence increasing the current density to facilitate explosion, is met.

(iii) The plate and the wires both are replaceable when the material gets exhausted.

The process of exploding the wire on the plate can take place using a DC current performing an instantaneous connection – disconnection. This is equivalent to using AC current source at a wide range of frequencies.

The present invention also discloses a portable apparatus for carrying out the explosion. The apparatus is both simple and efficient and resulted in production of large quantities of nanoparticles.

The wire guide (as shown in Figures 2 and 3) was prepared by the following method

(i) A one inch plastic rod of 4cm diameter was cut into two equal halves. Two perpendicular holes were drilled through one and the portion was cut open to fix a 5mm diameter glass bent tube (90° bent) in that place. This served as the wire guide through which the wire can be passed. This collimates the wire to go and strike the metal plate without bending in its way.

(ii) Two stainless steel plates fashioned into slides were stuck on the other half piece of the plastic block with an adhesive agent such as M-Seal™. The metal plate is easily slide-able through the stainless steel slides.

(iii) Hard plastic was used which is an insulator as well as it can withstand the explosion conditions.

(iv) A 500ml beaker was used as the vessel. Since it was required to keep the least distance between the metal plate and wire holder plastic block, handling it would definitely cause a problem – so the beaker was cut and its height was reduced to half.

Glass beaker cutting: A wire with its two free ends, each connected to a terminal of the battery was looped around the glass beaker along the mark at which the cut was to be done. Without touching the two ends, they were held tightly – then

current was passed through the wire. It became red hot and the glass beaker got cut along that mark.

(iii) The two plastic blocks were pasted with m-seal on the base of the beaker. Water was poured almost to the brim. The metal plate was passed through the slides on one block and the wire was passed through the tube attached to the other block placed opposite to the previous one.

(iv) Use of thick stranded copper wires with silver coating : This was used for the purpose of conduction from the battery's positive terminal to the metal plate by an ohmic connection and from the negative terminal to the wire by another ohmic connection to complete the circuit. The tip of the stranded wire was soldered. A hole was made below it so that a 4 to 5cm long thin etched wire could be looped in and this part was used for the explosion. One set of stranded wires was attached to the metal plate as well.

Dimension of the wire and the plate: Chemically etched wires were used for the purpose of explosion. The diameter was measured with the help of an optical microscope. A one inch by one inch metal plate was used. The following table discloses the diameter of the wires of different metals that were used for the electro-explosion of wires.

WIRE material	DIAMETER (μm) ²⁰
Iron	475
Silver	475
Copper	237
Aluminium	275

Etching of the metal plate: Before the plate could be used for the explosion, its surface should be chemically clean and smooth. For this the plate was etched. Etching was done in nitric acid in case of copper, nitric acid mixed with a few drops of hydrofluoric acid in case of iron and sodium hydroxide solution in case of aluminium. Silver wires of a required diameter were ordered. Nitric acid was poured in a beaker containing water to obtain a dilute solution. Then the plate was immersed in it and after sometime a clean metallic surface resulted. It was rinsed in distil water and dried.

The explosion was carried out for a range of voltages from 12 V to 48V. The minimum battery voltage available was 12 volts, so two and three such batteries were connected in series to meet the voltage requirements as needed.

Referring now to Figure 4, the electro-explosion of wires (EEW) is carried out employing a reaction vessel (1) prepared to house the electrodes (2, 2') and the medium (3) in which the explosion is carried out. The wire (2') is aligned to the correct geometry through the wire guide (5). The voltages required for the purpose of exploding the wire is provided by a bank of batteries (4) operating under the condition of an ideal current source. The plates (2) and wires (2') are cleaned through acid etching and the same method is employed to control the cross-section of the wires. The Al wires and plates were etched with a 10% solution of NaOH.

The background for the electro-explosion of wires has been presented by Graneau [P. Graneau, Phys. Lett. 97 A (1983) 253] who offers a theoretical explanation for this hitherto unexplained phenomenon, basing his argument on the axial forces expected to exist between separate elements of a current circuit according to Ampere-Neumann electrodynamics as they apply to steady currents. Graneau effectively specified an open circuit condition by which isolated circuit elements are subject to axial forces generated by mutual action of current in different parts of the element. The force is of the form:

$$F = i^2 \log (L/D)$$

Where (i) is the current in amperes and L/D is the ratio of the length of element L to a quantity D of the order of its thickness.

Graneau's emphasis that the fractures are tensile in character, whereas pinch forces are compressive and could not cause wire fragmentation of the form observed, but his theoretical account did not explain why the wire breaks into as many as 50 fragments. Each of these fragments is of insufficient length to develop adequate force.

According to Aspden [H. Aspden, *The exploding wire phenomenon*, Phys. Lett. 107 A, 5 (1985) 238], the most obvious cause for an axial force in the path of current flow is the direct action of electromotive force on charges in the conductor. When an emf is applied to a conductor the field intensity E applies force to the electron population having mobility, which accounts for current flow. The forces impart momentum to the electrons, which is transferred by collision forces are balanced by the action of E upon the positive charge of this atomic lattice. Overall, there is no resultant axial force on the conductor because the emf and the potential drop determined by the

collision related ohmic losses are balanced. Thus, in the steady state current condition, the closed circuital flow involves no axial forces along the current path.

Now, when the emf is changing owing to magnetic induction effects, including self-induction within the conductor, the applied emf and the potential drop are no longer in balance. Their difference can be measured experimentally and can account for an axial force in the line of current flow. Under these conditions the positive atomic lattice of the conductor is subject to the full intensity E , as are the electrons, but the electrons has an additional role. They not only act as a catalyst in transferring momentum to the lattice by collisions, but they also transfer momentum to whatever it acts as a store for the energy associated with the magnetic induction process. The field medium is closely coupled with the collective electron action and this field can assert forces in its interaction with charge in matter. In effect, therefore, we can reasonably expect a residual axial reaction force corresponding to the work done by the emf in feeding energy into the self-conductance of the conductor when its current increases. The force will be axial force acting between the conductor and the field induced in the observer's reference frame by the electron motion. Such a force can cause rupture of the conductor if the current build up is rapid enough, but it cannot separate the conductor body from the electron population. All that can be expected is that the conductor will disintegrate into elements, which are contained during the explosion within the plasma formed by the current discharge. The reason for this is that the force acting on each positive element of the atomic lattice of the conductor will not, in general, be the same throughout the conductor. The quantitative analysis fully supports this explanation. According to Graneau, the electrodynamics argument requires induced stress to be proportional to the square of current, whereas the new explanation suggested by Aspden [H. Aspden, *the exploding wire phenomenon as an inductive effect*, Phys. Lett. 120 A, 2, (1987) 80-82.] involves a linear current relationship.

The novelty and inventive step of the process of the patent resides in making metal nanoparticles dispersed in a dense medium through the electro-explosion of wires.

The following examples are given by way of illustration of the novel process of the present invention and therefore should not be construed to limit the scope thereof.

EXAMPLE - 1

A reaction vessel for preparation of nanomaterials employing the exploding wire technique was constructed out of glass with an arrangement for mounting a copper

metal plate (electrode 1) perpendicular to the base of the reactor. A wire guide arrangement is placed so that a copper wire (electrode 2), while passing through the guide, approaches the plate along its normal. The metal plate and the wire form the two electrodes which is connected to a battery bank allowing for supply of voltages starting at 12 V and going up to 48V in incremental steps of 12 V. The reactor vessel is filled up with a suitable dense medium so as to completely immerse electrode 2 and 66% of electrode 1.

The batteries were connected so as to have electrode 2 connected to the positive terminal of the battery and electrode 2' to the negative terminal. Electrode 2' is brought into contact with electrode 2 to achieve an explosion, following which the current naturally falls to zero. This signals the start of a new explosion sequence whereby the process is repeated.

The exploded metal particles remain suspended in the dense medium which is collected in the following manner. An initial centrifuge of the suspension at 5000 RPM separates the fluid from the solid mass. While the former is rejected, the solid mass is dispersed in electronic grade acetone for further AFM analysis. AFM analysis was carried out in the contact mode employing a silicon ultralever having a force constant of 0.2 N/m. The contact force was set at 10.4 nN for all topography data collected with the AFM. The nanoparticles dispersed in acetone was spread on single crystalline silicon (100). Figure 5 shows the AFM data collected for nano-crystalline copper particles allowing for the recording of particles in the range 27nm – 72nm. Part of the solid mass was incorporated in a paper matrix, dried and held firm for x-ray diffraction studies (XRD). First for reference an XRD pattern was generated for bulk copper as a θ - 2θ plot scanning from $41 - 100^\circ$ generating the lines (111), (200), (220), (311), (222) at $2\theta = 43.44^\circ$, 50.50° , 74.20° , 90.00° , and 95.10° respectively. For nano-copper sample collected at 36 V and incorporated in the said paper matrix, only one main peak at $2\theta=43.44^\circ$ was observed. Others peaks unlike those in the XRD pattern of bulk copper, were missing. This indicates the purity of the nanoparticle as far as the d - parameter relating to the (111) line is concerned. This further indicates reorientation of the nanoparticle grains preferentially in one direction as against the random orientation of grains in the bulk material. These details are shown in Figure 6.

EXAMPLE – 2

A reaction vessel for preparation of nanomaterials employing the exploding wire technique was constructed out of glass with an arrangement for mounting a silver

metal plate (electrode 2) perpendicular to the base of the reactor. A wire guide arrangement is placed so that a silver wire (electrode 2'), while passing through the guide, approaches the plate along its normal. The metal plate and the wire form the two electrodes which is connected to a battery bank allowing for supply of voltages starting at 12 V and going upto 48 V in incremental steps of 12 V. The reactor vessel is filled up with a suitable dense medium so as to completely immerse electrode 2 and 66% of electrode 1.

The batteries were connected so as to have electrode 1 connected to the positive terminal of the battery and electrode 2 to the negative terminal. Electrode 2' is brought into contact with electrode 2 to achieve an explosion, following which the current naturally falls to zero. This signals the start of a new explosion sequence whereby the process is repeated.

The exploded metal particles remain suspended in the dense medium which is collected in the following manner. An initial centrifuge of the suspension at 5000 RPM separates the fluid from the solid mass. While the former is rejected, the solid mass is dispersed in electronic grade acetone for further AFM analysis. AFM analysis was carried out in the contact mode employing a silicon ultralever having a force constant of 0.2 N/m. The contact force was set at 10.4 nN for all topography data collected with the AFM. The nanoparticles dispersed in acetone was spread on single crystalline silicon (100). Figure 7 shows the data collected for nano-crystalline silver particles allowing for the recording of particles in the range 50nm - 200nm.

Part of the solid mass was incorporated in a paper matrix, dried and held firm for x-ray diffraction studies (XRD). First for reference an XRD pattern was generated for bulk silver as a θ - 2θ plot scanning from 38° - 82° generating the lines (111), (200), (220), (311), (222) at $2\theta = 38.144^\circ$, 44.273° , 64.470° , 77.379° , and 81.500° respectively. For the nano-silver sample held onto a paper matrix as stated above, an XRD pattern was generated as a θ - 2θ plot scanning from 38° - 82° generating the lines (111), (200), (220), (311), (222) at $2\theta = 38.016^\circ$, 44.182° , 64.351° , 77.317° , and 81.500° respectively. The position of these lines in XRD is similar to those obtained in bulk silver. This indicate the purity of the nanoparticle lattice having bulk-like periodicity in the particles investigated. These details are shown in Figure 8.

EXAMPLE - 3

A reaction vessel for preparation of nanomaterials employing the exploding wire technique was constructed out of glass with an arrangement for mounting an iron

metal plate (electrode 2) perpendicular to the base of the reactor. A wire guide arrangement is placed so that an iron wire (electrode 2'), while passing through the guide, approaches the plate along its normal. The metal plate and the wire form the two electrodes which is connected to a battery bank allowing for supply of voltages starting at 12 V and going upto 48 V in incremental steps of 12 V. The reactor vessel is filled up with a suitable dense medium so as to completely immerse electrode 2 and 66% of electrode 1.

The batteries were connected so as to have electrode 1 connected to the positive terminal of the battery and electrode 2 to the negative terminal. Electrode 2 is brought into contact with electrode 1 to achieve an explosion, following which the current naturally falls to zero. This signals the start of a new explosion sequence whereby the process is repeated.

The exploded metal particles remain suspended in the dense medium which is collected in the following manner. An initial centrifuge of the suspension at 5000 RPM separates the fluid from the solid mass. While the former is rejected, the solid mass is dispersed in electronic grade acetone for further AFM analysis. AFM analysis was carried out in the contact mode employing a silicon ultralever having a force constant of 0.2 N/m. The contact force was set at 10.4 nN for all topography data collected with the AFM. The nanoparticles dispersed in acetone was spread on single crystalline silicon (100). Figure 9 shows the data collected for nano-crystalline iron particles allowing for the recording of particles in the range 10nm – 50nm.

Part of the solid mass was incorporated in a paper matrix, dried and held firm for x-ray diffraction studies (XRD). First for reference an XRD pattern was generated for bulk iron as a θ - 2θ plot scanning from 44° – 90° generating the lines (110), (200), (211) at $2\theta = 44.8^\circ$, 65.0° and 82.0° respectively. For the nano-iron sample held onto the paper matrix as stated above, an XRD pattern was generated as a θ - 2θ plot scanning from 44° – 90° generating the lines (110), (200), (211) at $2\theta = 44.8^\circ$, 65.0° , and 82.0° respectively. The position of these lines in XRD is similar to those obtained in bulk iron. This indicates the purity of the nanoparticle lattice having bulk-like periodicity in the particles investigated. These details are shown in Figure 10.

EXAMPLE – 4

A reaction vessel for preparation of nanomaterials employing the exploding wire technique was constructed out of glass with an arrangement for mounting an aluminium metal plate (electrode 2) perpendicular to the base of the reactor. A wire

guide arrangement is placed so that an aluminium wire (electrode 2'), while passing through the guide, approaches the plate along its normal. The metal plate and the wire form the two electrodes which is connected to a battery bank allowing for supply of voltages starting at 12 V and going upto 48 V in incremental steps of 12 V. The reactor vessel is filled up with a suitable dense medium so as to completely immerse electrode 2 and 66% of electrode 1.

The batteries were connected so as to have electrode 1 connected to the positive terminal of the battery and electrode 2 to the negative terminal. Electrode 2 is brought into contact with electrode 1 to achieve an explosion, following which the current naturally falls to zero. This signals the start of a new explosion sequence whereby the process is repeated.

The exploded metal particles remain suspended in the dense medium which is collected in the following manner. An initial centrifuge of the suspension at 5000 RPM separates the fluid from the solid mass. While the former is rejected, the solid mass is dispersed in electronic grade acetone for further AFM analysis. AFM analysis was carried out in the contact mode employing a silicon ultralever having a force constant of 0.2 N/m. The contact force was set at 10.4 nN for all topography data collected with the AFM. The nanoparticles dispersed in acetone was spread on single crystalline silicon (100). Figure 11 shows the data collected for nano-crystalline aluminium particles allowing for the recording of particles in the range 40nm - 150nm.

Part of the solid mass was incorporated in a paper matrix, dried and held firm for x-ray diffraction studies (XRD). First for reference, an XRD pattern was generated as a $\theta - 2\theta$ plot for bulk aluminium scanning from $40^\circ - 85^\circ$ generating the lines (200), (220), (311), (222) at $2\theta = 45.2^\circ, 65.4^\circ, 78.4^\circ$ and 82.6° respectively. For nano-aluminium the first peak was observed at $2\theta = 46.1^\circ$ and the second peak was observed at $2\theta = 66.8^\circ$. No peaks were observed after that. These peaks were comparatively broadened with respect to those of bulk aluminium. The position of these lines for the nano-aluminium are distinct from those of the bulk material indicating a highly distorted aluminium lattice in the nanomaterial. These details are shown in Figure 12.

The main advantages of the novel process of the present invention are:

1. Nanoparticles from conducting parent materials which can be shaped in the form of a wire/plate can be used. In this case both electrodes produce the particles.
2. The dominant mechanism here is spark explosion, which is an adaptation of the electro-explosion of wires phenomena.

3. Our voltages are much lower 12V – 48V maximum. We can also use AC currents for uniformity, according to theory.

4. We can use many dense media such as glycerin, hydrocarbon oil etc. as polarizability of the media is of no importance.

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